

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. § 371		Attorney's Docket Number 056118-5001
International Application. No.	International Filing Date	U.S. Application No. Unassigned
PCT/US00/20781	August 16, 2000	Priority Date Claimed August 17, 1999
Title of Invention: PROCESS FOR REMOVAL OF UNDESIRE FLAVOR FROM ENZYME-HYDROLYZED OAT BRAN AND THE RESULTING PRODUCT		
Applicant For EO/EO/US: Arthur P. HANSEN		

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a FIRST submission of items concerning a filing under 35 U.S.C. § 371.
2. ☐ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. § 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. § 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. § 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. § 371(c)(2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. § 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. § 371(c)(3)).
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. § 371(c)(3)).
9. ☐ An oath or declaration of the inventors (35 U.S.C. § 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. § 371(c)(5)).

**Items 11. to 14. below concern other document(s) or information included:**

11. ☒ An Information Disclosure Statement under 37 C.F.R. § 1.97 and § 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. § 3.28 and § 3.31 is included.
13. ☐ A FIRST preliminary amendment.
14. ☒ A SECOND or SUBSEQUENT preliminary amendment.
14. ☒ Other items or information:
  - a. PCT/IB/304
  - b. PCT/IB/308
  - c. PCT/IPEA/409



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PROCESS FOR REMOVAL OF UNDESIRE  
D FLAVOR FROM ENZYME-HYDROLYZED  
OAT BRAN AND THE RESULTING PRODUCT

5

The present invention relates primarily to a process for the removal of undesired oat flavor from enzyme hydrolyzed oat bran and to the resulting product. The product is particularly useful as an addition to food products,  
10 notably dairy products, to improve health qualities.

Background of the Invention

Enzymatically hydrolyzed oat bran products, e.g. the product "Oatrim", are well known. Such products are obtained by the process described in U.S.  
15 Patent Nos. 4,996,063 and 5,082,673. Briefly, the process comprises gelatinizing a mixture of oats and water by passage through a steam injection cooker at a temperature of between 138°C to 143°C. The pH of the mixture is then adjusted and alpha-amylase enzymes are added so as to hydrolyze the starch in the mixture. After the hydrolyzation is completed, soluble fiber is  
20 separated and dehydrated to give the "Oatrim" product. This can be added to dairy products, e.g. diet drinks, milk or frozen confections. Alternatively, the hydrolyzed oat bran product can be used to produce such products as cheese, yogurt, etc.

The use of "Oatrim" or the like as a food additive is extensively  
25 described in the literature. See, for example, U.S. Patent Nos. 5,723,162; 5,709,900 (low fat cheese); 5,626,849 (weight loss composition); 5,607,716 (low fat confectionery items); 5,585,131 (meat products); and 5,532,018 (low fat cheese). The product is considered to be particularly useful as a heart healthy food additive in that it has been shown to provide a substantial drop in  
30 artery-clogging LDL cholesterol without substantial drop in beneficial HDL cholesterol. An article by Raloff (Food Technology, 1991, Vol. 8, beginning at page 62) provides more information as to the health benefits which are realized by consuming enzymatically hydrolyzed oat bran product.

While hydrolyzed oat bran products such as "Oatrim" are described as  
35 essentially taste-free, a limiting factor in the use of such products as food additives has been the fact that, as currently available, these products tend to

retain an oat taste that the public finds generally unacceptable. As a consequence, considerable research effort is being directed towards providing such products which are free from any oat taste.

## 5 Summary of the Invention

The principal object of the present invention is to provide a process for treating "Oatrim" or like enzymatically hydrolyzed oat bran product so as to remove undesirable oat taste while retaining other desired characteristics.

Another object is to provide improved food products, for example, milk  
10 and related dairy products containing an oat-based product according to the invention which are characterized by freedom from any undesired oat taste while providing the health advantages attributable to "Oatrim" and similar products.

Broadly described, the present process comprises subjecting  
15 hydrolyzed oat bran, e.g. "Oatrim", to extraction with a lower alkanol followed by removal of the alkanol. It has been found that product free from undesired oat taste and otherwise taste-free can be obtained in this way. As a consequence, the product can be added to, for example, milk or other foods to provide a product which is free of any oat taste while otherwise maintaining  
20 all of the healthful characteristics of "Oatrim" or equivalent hydrolyzed oat bran product.

## Description of Preferred Embodiments

For ease of reference, enzymatically hydrolyzed oat bran, such as  
25 "Oatrim" made according to the aforementioned U.S. Patents 4,996,063 and 5,082,673, is generally referred to herein as "oat flour".

Preferred features of the invention are described hereinafter in conjunction with the accompanying drawings wherein:

Figure 1 represents a flow diagram illustrating a continuous extraction  
30 process according to the invention;

Figure 2a diagrammatically illustrates a system for drying of the extracted oat flour and recovery of the alcohol used in the extraction;

Figure 2b is a side view of a rotary vacuum drier suitable for use in the invention;

Figure 2c is an end of the drier shown in Figure 2b;

Figure 3 diagrammatically illustrates the grinding of the oat flour which has been processed according to the invention; and

Figure 4 diagrammatically illustrates another continuous system for  
5 carrying out the process of the invention.

Referring more specifically to the drawings, the system shown in Figure 1 comprises an extraction vessel (2) of stainless steel or the equivalent in which there are placed bags (4) containing the oat flour. The bags are made of material which is of sufficiently fine mesh to retain the oat flour while open  
10 enough to permit the solvent to pass therethrough.

Advantageously, the bags rest on a stainless steel screen or grate (5) positioned above the outlet of the vessel. This facilitates removal of the solvent from the extractor as noted below.

Means, for example, a steam jacket (7), are provided for heating the  
15 contents of the vessel.

Solvent, which is preferably 95% ethanol although other lower alkanol or the equivalent can also be used, is fed to the top of the extraction vessel (2) via conduit (6). The solvent is pulled through the bags (4) within the extraction vessel by means of a Waukesha positive pump (8) or the like which  
20 is positioned at a point below the outlet (10) of the extraction vessel. The pump then forces solvent withdrawn from the extraction vessel to an ultra filtration membrane (12) or the like which removes from the solvent some of the material extracted from the oat flour. As an example, the ultra filtration membrane may be selected to remove extracted matter having a molecular weight in excess of 200, e.g. 200-400. The use of the ultra filtration  
25 membrane or equivalent is optional and this feature may be eliminated, particularly where the matter extracted from the flour has a molecular weight below 200.

The solvent then leaves the ultra filtration membrane (12), if used, via  
30 conduit (14) for a charcoal filter (16) or the like to remove any color and/or flavor substances with a molecular weight of less than 200. The thus processed solvent may then be passed via (6) back to the extraction vessel (2). Makeup solvent, if needed, can also be added at this stage or at some other convenient point in the system.

Preferably, at least some, and possibly all, of the solvent withdrawn from the extraction vessel is passed into a chiller (22) where the solvent is cooled to crystallize and remove fats or lipids extracted from the oat flour. A temperature in the range of 34°F to 40°F, preferably about 36°F, may be employed for the chiller. Usually, the solvent will be kept in the chiller for 1 to 2 hours to enable removal of the fats or lipids in crystal form.

Overall, the extraction process as shown in Figure 1 may be completed in about 20-24 hours, with solvent preferably being continuously circulated through the system. In an alternative arrangement, the process may be carried out batch-wise. In such case, the oat flour is allowed to stand in extraction vessel (2) steeped in solvent which is periodically withdrawn and replaced every 8 hours with fresh solvent. Whether carried out continuously or batch-wise, the solvent used for the extraction is advantageously processed for further use as described above by, for example, chilling to remove fats and passage through suitable filter means or by distillation to remove color and flavor substances and/or other contaminants before the solvent is re-used.

The extraction is preferably carried out at elevated temperature and pressure. Temperatures in the range of 140°F to 160°F and pressures of about 3 to 7 psig are preferred. However, other temperatures and pressures may be used.

Residence time in the extraction vessel will vary depending on other conditions, e.g. the amount of oat flour being processed, whether or not the process is carried out continuously or batch-wise, etc. Usually, however, a total time of 20-24 hours contact time between the oat flour and solvent is sufficient for the extraction. For batch operations, several extraction cycles of, for example, 8 hours each can be employed as earlier noted. Usually three such cycles are sufficient to provide the desired taste-free product. In continuous operations, the solvent can be continuously fed into the vessel and removed therefrom, the process being continued until the oat flour is taste-free.

Following the extraction with alkanol, the processed oat flour must be dried to remove all of the alcohol. This may be done in various ways, e.g. by allowing the oat flour to dry in air and/or by placing the oat flour in a heated

drier such as a vacuum drier, operable at a temperature (e.g. 120 - 130°F) sufficient to volatilize the alkanol without damaging the oat flour.

Figures 2a -2c illustrate one way of drying of the oat flour after extraction. As shown, the oat flour, after the extraction, is placed in a rotary vacuum drier (24) or the equivalent where much of the alcohol solvent (e.g. up to 99% or more) is recovered. In the embodiment shown, the drier, which may be in the form of a rotatable screened drum, is positioned within a sealed vessel (25) or the like so that the alcohol, as it is evaporated in the rotary vacuum drier, passes via an appropriate conduit (26) into a chilled condenser (28) where the alcohol is condensed. This alcohol can then be fed into an appropriate storage tank (30) for later use or, if desired, the condensed alcohol can be fed directly to the extraction vessel for use.

As schematically shown in Figures 2b and 2c, the vacuum drum (24) is rotatable about a horizontal axis (27) which is itself rotatable on supports (29), the volatilized alcohol escaping from the drum through small screen openings (31).

The solvent-free oat flour is then further processed in, for example, a stainless steel Fitzmill grinder (34) as shown in Figure 3 to provide a desired particle size, e.g. 20-80 mesh. The ground product is then placed into suitable containers, e.g. polyethylene bags, which should be heat sealed. The packaged product is ready for incorporation into, for example, dairy foods, nutritional drinks or frozen desserts, to provide the health advantages of "Oatrim" or the like but without any oat flavor.

The invention is further described by reference to the following examples:

#### Example 1

Fresh "Oatrim" (oat flour) was placed in eight sterile muslin bags (4), using 30 pounds per bag. The bags were tied off with colorless twine. The bags were then placed in a clean stainless steel pressurized-jacketed extractor (2). The bags were positioned on a stainless steel screen or grate (5) a distance, e.g. 18 inches, above the drain line or outlet (10). This made it possible to pump the extractant (95% ethanol) out of the vessel without blocking the suction side of the pump (8).

After the eight bags were placed in the extractor, a gasket around the open top of the extractor (not shown) was lubricated and a lid (11) was placed over the extractor top, closed and sealed. Cap bolts (not shown) were tightened across from each other to tighten the lid evenly.

5            Steam was then turned on to heat the jacketed extractor (2) to 160°F and the pump (8) was turned on to begin circulating the alcohol over the oat flour. The hot alcohol was pulled through the bags of oat flour to remove the oat flavor and color. This extraction was continued for 12 hours under heat of 160°F and pressure (6 psi). After 12 hours, the heat was shut off along with  
10   the pump and the mixture was allowed to cool to room temperature (70°F).

         The next day, the top of the extractor (2) was opened to allow air in so that the alcohol extract could be pumped into a collection drum or drums. Approximately 75 gallons of ethanol (95%) were pumped into the extraction with a yield of 45 to 50 gallons pumped out after 24 hours. This difference  
15   between the amount of alcohol fed in and taken out was due to the 240 pounds of "Oatrim" absorbing 25 to 30 gallons of ethanol. The above extraction was repeated two further times. The second extraction only used approximately 55 gallons of ethanol (95%). This was pumped into the extractor and the extractor lid was sealed and heat turned on to 160°F as  
20   before. The positive pump (8) was started and the hot alcohol was circulated over the bags of "Oatrim" to remove oat flavor. The alcohol was circulated for 12 hours at 160°F under pressure (6 psi). After the 12 hours, the extractor was shut off and allowed to cool overnight to room temperature. The lid was then opened and the alcohol extract was again pumped into collection drums.

25           For the third and final extraction, 55 gallons fresh ethanol (95%) were pumped into the extractor (2), the lid was closed and the alcohol heated to 160°F. The ethanol was circulated through the oat flour for another 12 hours. The extractor was then shut off and allowed to cool down to room temperature. The next day, the extractor lid was opened and the alcohol was  
30   pumped out into the collection drum. The bags of extracted "Oatrim" were then removed from the extractor and placed in stainless steel pans to air dry. The extracted product was stirred three to four times per day to accelerate the drying. The final drying was done in a convection oven at 130°F to prevent



any chemical alteration to the product due to Maillard Reaction or protein denaturation. Once the mixture was totally dried of the ethanol, the product was ground to eighty mesh in a stainless steel Fitzmill grinder (34) as shown in Figure 3. The product, which was free of taste and odor, was then packaged in multi-layered polyethylene bags for use in the fortification of fat-free milk and chocolate milk.

### Example 2

Two hundred and forty pounds oat flour ("Oatrim") was extracted with seventy-five gallons of 95 % ethanol for twelve hours at 140-160°F. This was followed by a second extraction using sixty gallons of 95 % ethanol for twelve hours at the same temperature. A third extraction then followed using sixty gallons of fresh ethanol and extracting for twelve hours at 140-160°F. The extracts contained color and oat flavor. The color of the first extract was similar to weak tea whereas the second extraction was lighter and the third similar to off-colored water.

Coconut charcoal was ground up to be granular packed in a glass column with glass wool on the top and bottom of the charcoal bed. The oat extract from each extraction was passed through the coconut charcoal at a slow rate. This removed all of the color and the flavor from the ethanol rendering it colorless and free of oat flavor. This process was repeated on each extract from the first, second and third extractions. To further test the viability of using the charcoal to clean up the ethanol for further use, a gallon of the first extraction was concentrated from one gallon to sixteen ounces and this concentrate was then passed through a fresh coconut charcoal filter with the same results, i.e. the filtered ethanol was colorless and flavorless as related to oat flavor.

### Example 3

Further testing was done on a commercial scale using a stainless steel housing with a charcoal filter placed in the housing. For this purpose, the oat flour was put in muslin bags tied with nylon rope and placed in the extractor as shown in Figure 4. Seventy-five gallons of ninety-five 95 % ethanol were

then pumped on top of the oat flour. The lid of the extractor was greased and sealed with the nuts cranked down on the opposite sides until completely sealed. The ethanol was heated to 140-160°F and circulated with a Waukesha pump (8) through the charcoal filter 35 and into the extractor (2) as shown in Figure 4. The extractor was run for eight hours continuously. After the eight hour run, the extractor was shut down, allowed to cool overnight and a sample of the oat flour was removed and analyzed in skim milk for oat flavor. There was a slight oat flavor in the milk. The extractor was then run continuously for another eight hours and a sample of oat flour randomly removed from one of the oat flour bags in the extractor. The sample was dried, sifted and placed in skim milk and tested for oat flavor. At this time, eight hours to be certain that all of the oat flavor was totally removed from the oat flour. The sample was dried, sifted and placed in skim milk for flavor evaluation. There was no oat flavor remaining in the oats.

This example indicates that in the arrangement shown in Figure 4 using a charcoal filter, the oat flour can be effectively processed in twenty-four hours to remove all of the oat flavor while allowing the same alcohol to be used over and over again throughout the process. This has the dual advantage of reducing the overall time involved in processing the oat flour into a taste-free state while at the same time reducing the loss of alcohol which would otherwise occur. Charcoal filters appear to be particularly useful for this purpose as ultra filtration membranes or millipore filters do not appear to be as effective, particularly with respect to removing extracted matter at the 200 – 400 molecular weight level.

It will be appreciated that various modifications may be made above. Thus, while the invention has been illustrated above by reference to the processing of "Oatrim", it will be recognized that the invention is broadly useful towards improving the characteristics of other types of oat flour products made by enzymatically hydrolyzing oat bran or the like. Furthermore, while the invention has been described in the foregoing with respect to the processing of oat flour, it is contemplated that the invention can also be used to remove undesirable flavors from soy flour and the like by use of the process described herein.

[illegible]

I claim:

1. A process for removing oat flavor from oat flour which comprises extracting the oat flour with a lower alkanol until the oat flour is free of oat flavor.
2. The process of claim 1 wherein the lower alkanol is ethanol.
3. The process of claim 2 wherein the extraction is carried out at elevated temperature and pressure.
4. The process of claim 1 wherein the oat flour after extraction is vacuum dried to remove any traces of the lower alkanol.
5. The process of claim 1 carried out continuously.
6. The process of claim 1 carried out batch-wise.
7. The process of claim 1 wherein the alkanol, after extracting the oat flour, is processed to remove lipids, filtered to remove any color and flavor compounds extracted from the oat flour and then recycled for further extraction of the oat flour.
8. The process of claim 1 wherein the extraction with lower alkanol is carried out for 20-24 hours at elevated temperature and pressure.
9. The process of claim 1 wherein the oat flour is "Oatrim".
10. The product obtained by the process of claim 1.
11. A process for removing soy flavor from soy flour which comprises extracting the soy flour with a lower alkanol until the soy flour is free of soy flavor.



## ABSTRACT OF THE DISCLOSURE

A process for removing flavor from hydrolyzed oat bran which comprises extracting the hydrolyzed oat bran with a lower alkanol until the oat bran is free of oat taste. The corresponding processing of soy flour is also disclosed.

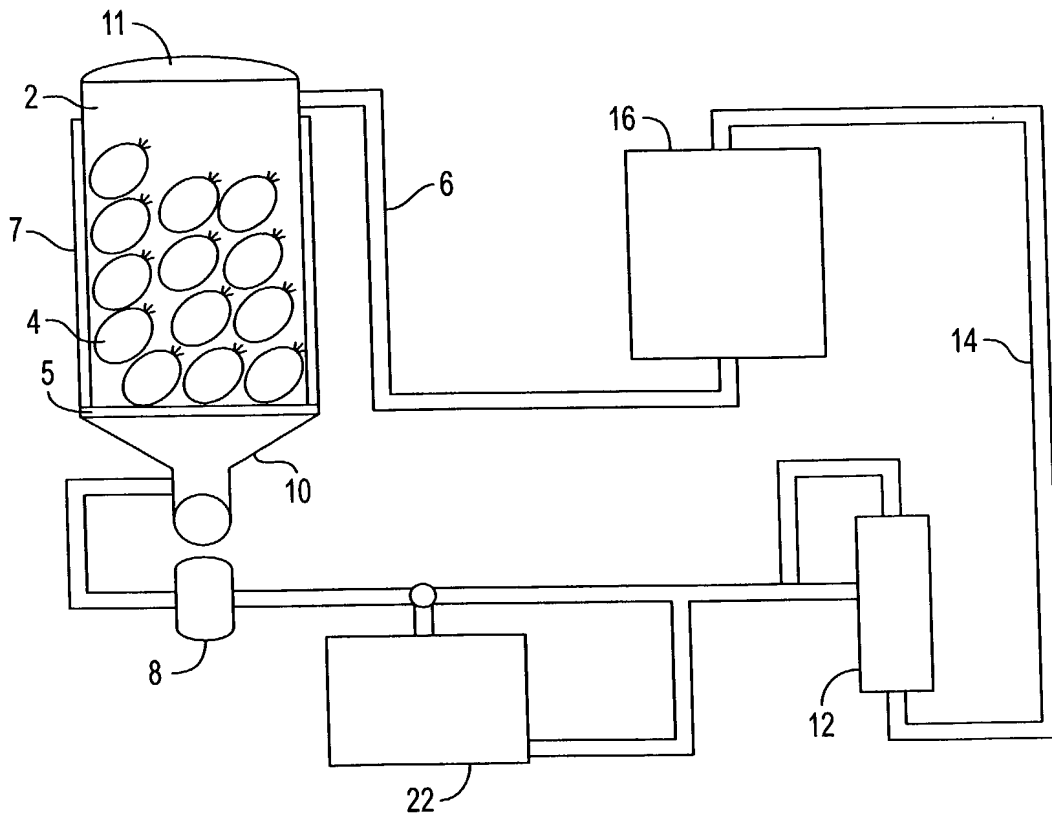


FIG. 1

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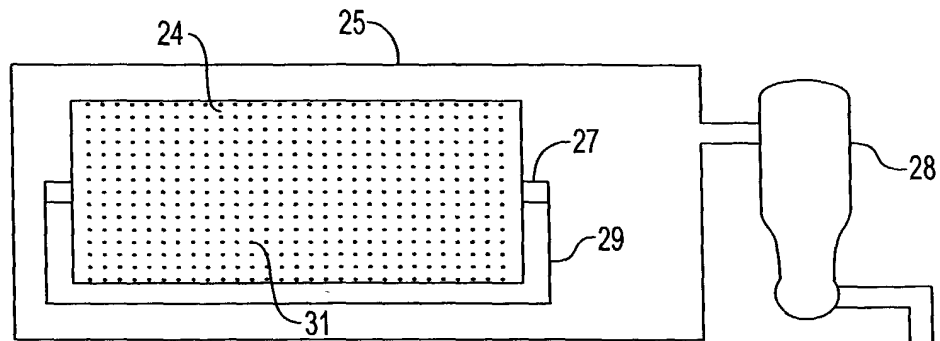


FIG. 2A

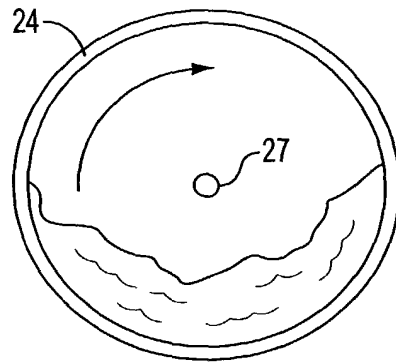


FIG. 2C

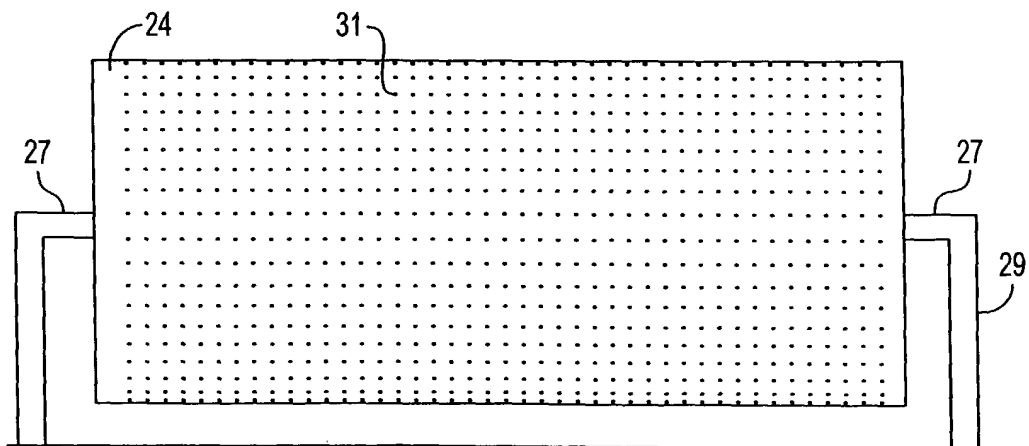
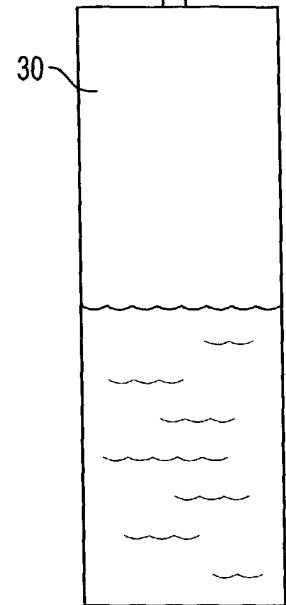


FIG. 2B

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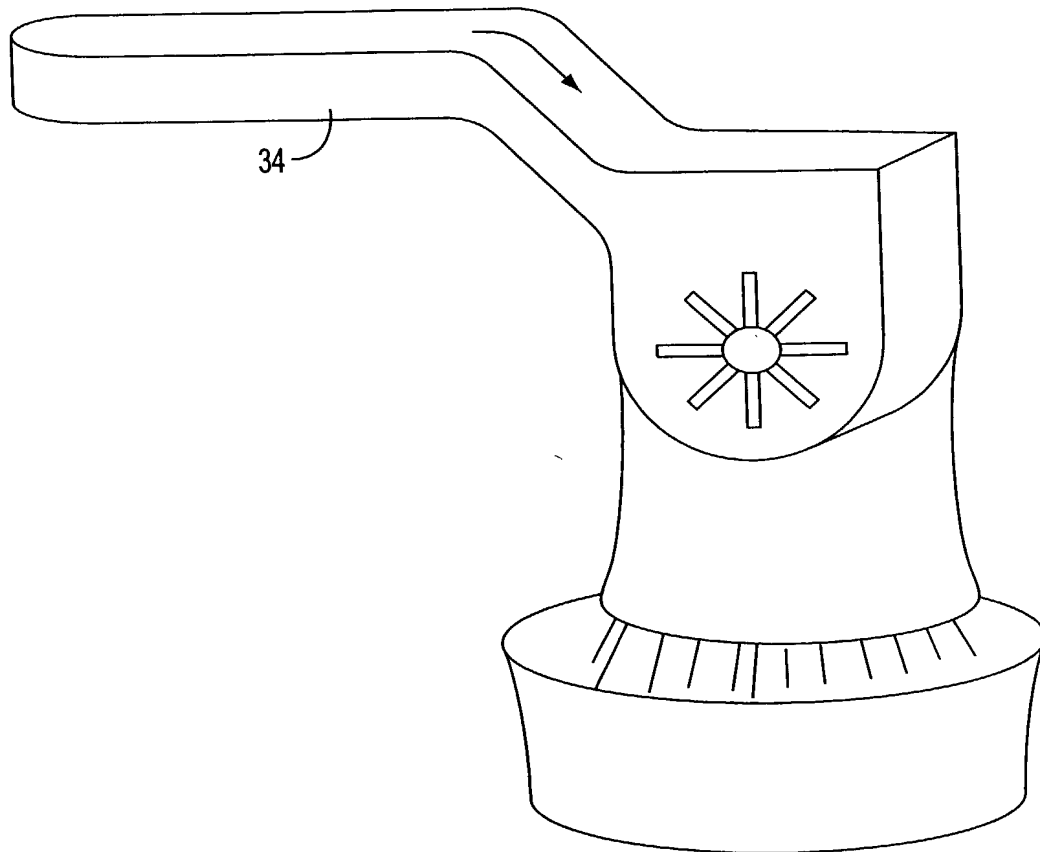


FIG. 3



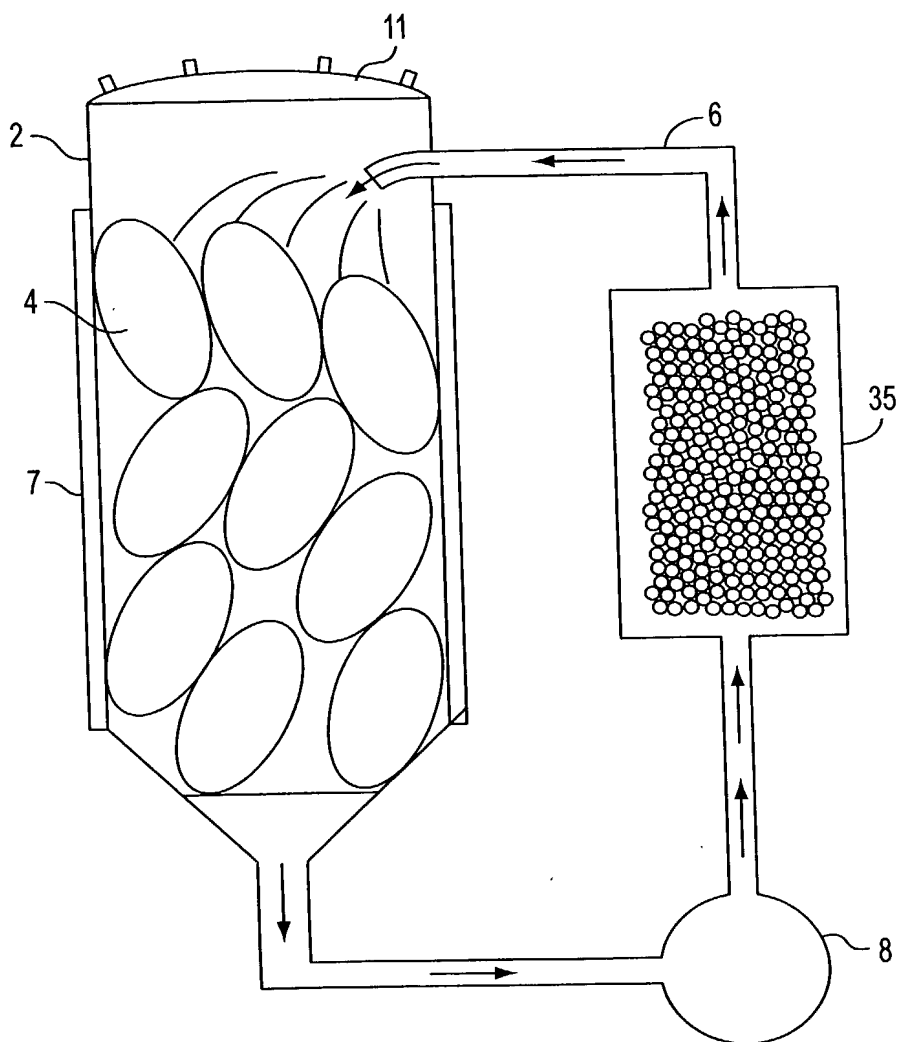


FIG. 4

**COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY**U.S. DEPARTMENT OF COMMERCE  
Patent and Trademark Office

ATTORNEY DOCKET NO.: 056118-5001

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**PROCESS FOR REMOVAL OF UNDESIRE FLAVOR FROM ENZYME-HYDROLYZED OAT BRAN AND THE  
RESULTING PRODUCT**

The specification of which:

is attached hereto; or

was filed as United States application Serial No. 10/049,833 on February 19, 2002 and was amended on \_\_\_\_\_ (if applicable); orwas filed as PCT international application Number PCT/US00/20781 on August 16, 2000 and was amended under PCT Article 19  
On \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the U.S. Patent and Trademark Office information which is material to the patentability of claims presented in this application in accordance with Title 37, Code of Federal Regulations Section 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) or Section 365(b) of any foreign application(s) for patent or inventor's certificate or Section 365(a) of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign applications(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed.

**PRIOR FOREIGN APPLICATION(S):**

COUNTRY (if PCT, indicate PCT)	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED	
			Yes	No
			<input type="checkbox"/> Yes	<input type="checkbox"/> No
			<input type="checkbox"/> Yes	<input type="checkbox"/> No
			<input type="checkbox"/> Yes	<input type="checkbox"/> No

**Combined Declaration for Patent Application and Power of Attorney - (Continued)**  
 (includes Reference to PCT International Applications)  
**ATTORNEY DOCKET NO.: 056118-5001**

I hereby claim the benefits under Title 35, United States Code Section 119(e) of any United States provisional application(s) listed below.

**U.S. PROVISIONAL APPLICATIONS**

**U.S. PROVISIONAL APPLICATION NO. 60/149,246      U.S. FILING DATE: August 17, 1999**

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) or Section 365(c) of any PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, Section 112, I acknowledge the duty to disclose to the U.S. Patent and Trademark Office all information known to me to be material to the patentability of claims presented in this application in accordance with Title 37, Code of Federal Regulations, Section 1.56 which became available between the filing date of the prior application(s) and the national or PCT international filing date of this application:

**PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT:**

U.S. OR PCT INTERNATIONAL APPLICATIONS		STATUS (Check One)		
APPLICATION NO.	FILING DATE	PATENTED	PENDING	ABANDONED
PCT/US00/20781	16 Aug 2000	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

**POWER OF ATTORNEY:** As a named inventor, I hereby appoint the registered practitioners of Morgan, Lewis & Bockius LLP included in the Customer Number provided below to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith, and direct that all correspondence be addressed to that Customer Number.

Customer Number: **09629**

Direct Telephone Calls To:


**Paul N. Kokulis**  
**202-739-5455**

Combined Declaration for Patent Application and Power of Attorney - (Continued)

(includes Reference to PCT International Applications)

ATTORNEY DOCKET NO.: 056159-5003

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

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FIRST OR SOLE INVENTOR'S SIGNATURE 	DATE 6/26/02	
FULL NAME OF SECOND INVENTOR		
RESIDENCE & CITIZENSHIP		COUNTRY OF CITIZENSHIP
POST OFFICE ADDRESS		
SECOND INVENTOR'S SIGNATURE	DATE	
FULL NAME OF THIRD INVENTOR		
RESIDENCE & CITIZENSHIP		COUNTRY OF CITIZENSHIP
POST OFFICE ADDRESS		
THIRD INVENTOR'S SIGNATURE	DATE	

Listing of Inventors Continued on attached page(s): ☐ Yes ☐ No